



Electrocoagulation as a possible treatment for wastewater polluted with industrial lubricant oils

La electrocoagulación como posible tratamiento para aguas residuales contaminadas con aceites lubricantes industriales

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Abstract

Electrocoagulation has demonstrated significant removal effectiveness of grease, oils, biodiesel and microplastics in wastewater. The optimum operation parameters for industrial lubricant oils removal in wastewater were defined in this investigation by using a parallel array of two pair of aluminum 6061 electrodes. The experiment was performed using a rectangular batch reactor made up of glass where an analogous water polluted with 1 ml of industrial lubricant oil waste was treated applying a current density of 40 A/m² in a range of 10 to 50 minutes and varying the addition of 100 ml of a 2 g/L NaCl solution. The optimum operating conditions within the range analyzed achieved 91% of removed lubricant after 30 minutes of treatment and using the electrolyte additive. Furthermore, the analogous treated water contained 1.6 mg/L of Total Suspended Solids (TSS), 8.44 pH units, 0.97 Nephelometric Units of Turbidity (NUT) and 14.33 color units in the platinum/cobalt scale. These results make the treated water a possible candidate as service or irrigation water.

Keywords: electrocoagulation, lubricant oils, removal, treatment, wastewater.

Resumen

La electrocoagulación ha mostrado resultados efectivos para la eliminación de grasas, aceites, biodiesel y microplásticos en aguas residuales. En la presente investigación se definieron las condiciones de operación óptimas para la remoción de aceites lubricantes industriales presentes en aguas residuales utilizando un arreglo en paralelo de dos pares de electrodos fabricados en aluminio 6061. El experimento se realizó en un reactor batch rectangular en donde se trató un agua residual análoga contaminada con 1 ml de residuos de aceite lubricante industrial aplicando una densidad de corriente de 40 A/m² en intervalos de 10 a 50 minutos y variando la adición de 100 ml de solución de NaCl a 2 g/L. Las condiciones óptimas alcanzaron hasta un 91% de aceite removido con un tiempo de operación de 30 minutos, una densidad de corriente de 40 A/m² y utilizando una solución de 2 g/L como electrolito soporte. Asimismo, el agua análoga tratada presentó 1.6 mg/L de Sólidos Suspendidos Totales (SST), 8.41 unidades de pH, 0.97 Unidades Nefelométricas de Turbidez (UNT) y 14.33 unidades de color verdadero en la escala platino cobalto (Pt/Co). Estos resultados le confieren una calidad apta su uso humano.

Palabras clave: agua residual, aceite lubricante, electrocoagulación, remoción, tratamiento.

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1 Introduction

Industrial lubricant oils are special substances that aim to reduce the strong friction effects over two surfaces in contact during the mechanical movement of all scale machinery, such as engines. These products are also able to deal with physical impurities since they can perform as anticorrosive substances, power transmission media, cooling agents or sealers. The usage of lubricant oils allows to keep all the equipment and tools in their optimal conditions promoting corrective maintenance savings (Quesada, 2021). Thanks to these excellent properties, lubricant oils market surpassed 800 million liters per year in Mexico during 2015 (Portal Automotriz).

Regarding the formulation of the lubricant oils, it is possible to identify two main components that are present in a variable mix ratio: oily base and additives. The oily base represents the largest part of the product and determines the main properties of the lubricant. The additives provide or adequate these properties to the needs of the different industries e.g., automotive, energy, oil and gas, mining, metal mechanic or manufacturing sector (Sanz, Andrade, 2015).

Due to the extent lubricant oils market, large amounts of oil waste are produced not only in Mexico but all around the globe. This waste is generated from the replacement of all the old lubricant oils with a deficient performance because of its age or impurity content. This kind of waste is hazardous for both the environment, wildlife, and public health due to their high content in aromatic hydrocarbon compounds and metals such as chromium, cadmium, or lead (Fong, 2017). As defined by Jurado, 2017, it only takes one liter of used industrial lubricant oil to pollute a thousand liters of clean water. In consequence, the discharge of this hazardous waste in aquatic systems has a negative impact over the environment, especially when filtered to groundwater. This filtration may cause aquatic species death and ground pollution threatening the environment and human health induced by the usage and contact with polluted water.

Industrial lubricant oils are considered as hazardous waste after its usage according to the Mexican law that legislate the disposal and management of any kind of waste *Ley General para la Prevención y Gestión Integral de Residuos* since they present flammable and toxic properties. A wrong management of industrial lubricant oils waste may lead to their presence in sewage water that reaches aquatic ecosystems. The more volume of wastewater polluted with oils is produced, the more important becomes to explore the viability of approachable, environmentally friendly, and effective technologies of oily wastewater treatment.

In recent years several research have been developed regarding a possible application of the electrocoagulation technique in wastewater containing a wide variety of polluting agents, such as metals, organic compounds, dyes, microplastics, biodiesel, cooking oils and organic material

(Moussa *et al.*, 2015). The results are promising as electrocoagulation has demonstrated a significant removal effectiveness of this hazardous waste and the fact that produces less amount of sludge because no additional chemical compounds are needed, avoiding further difficult separation processes (Perozo, 2017; Morales, 2015; Gobb, 2018; Morales, 2018; Perren *et al.*, 2018; Changmai *et al.*, 2019; Rodrigues *et al.*, 2015).

Electrocoagulation process is performed in an electrochemical reactor that includes aluminum or steel-made electrodes and direct current (DC) is supplied through the system, that induces the oxidation process of the anodes where metallic hydroxide species are formed, such as Al^{3+} or Fe^{2+} , depending on the base material of the electrodes (Gobb *et al.*, 2018). These ionic species destabilize the electrical charges of the emulsion, promoting the formation of flocs that can be easily removed by filtration. On the other hand, the reduction of the water in the cathode produces hydrogen gas that induces movement within the water matrix and the further flotation of the flocs to the surface of the system when using aluminum electrodes (An *et al.*, 2017).

A deep documental review of the latest research allowed to determine the critical operating parameters that have a strong effect over the operation and removal effectiveness of electrocoagulation technique and thereby to set the appropriate limits of the value ranges that were more likely to produce the desired removal effect on a system polluted with a sample of industrial lubricant oil waste.

Therefore, an experimental study consisting of ten characterization trials was conducted to determine the optimum operating conditions to achieve the largest amount of lubricant oil waste recovered applying electrocoagulation to a wastewater analogue as an electrochemical removal treatment. It is worth to mention that characterization trials were conducted at a constant value of current density of 40 A/m² that, according to preliminary trails included in the study, demonstrated to be the appropriated value considering the generation speed of floccules where significant results are obtained in the range from 18 to 60 A/m² (Rodrigues *et al.*, 2015).

2 Materials and methods

2.1 Equipment description

The wastewater analogue was treated using electrocoagulation in a rectangular batch reactor to allow a better distribution of the aluminum 6061 electrodes (Perren *et al.*, 2018). The dimensions of the batch reactor were 9" large, 6' height and 4.7" width. On Figure 1, the electrocoagulation equipment is shown, including the batch reactor which was made up of glass to facilitate the observation of the electrocoagulation process, as well as the evolution of the analogue wastewater changes in terms of

color and turbidity. The aluminum 6061 electrodes had 3/8" thick, 3" large and 2" width to enhance bubbles formation by the increased contact area between the analogue wastewater and the electrodes. Aluminum 6061 was selected as electrode material based on the recommendations to prevent water orangeness made by Morales, 2015.

As Figure 2 shows, the electrodes were connected in a monopolar parallel arrangement using electrical black and red wiring to identify negative and positive electric flux, respectively. Besides, the monopolar parallel arrangement was selected to avoid reduction of the removal efficiency caused by formation of oxides in the electrodes surface (Moussa *et al.*, 2015).

Energy for electrocoagulation process was provided by a power supply with a capacity of 32 V and 5 A of Direct Current (DC). The DC intensity and voltage were monitored by connecting a multimeter to the electric circuit composed by the two pairs of aluminum 6061 electrodes immerse in the wastewater analogue. Furthermore, the multimeter eased DC supply control by showing real time values. Moreover, a potentiometer was employed to measure water pH before treatment. This parameter was not manipulated during the experimental study to avoid hazardous chemicals such as strong acids or bases commonly used as buffer solutions. Nevertheless, initial pH values were registered as a reference to perceive any change after electrocoagulation treatment.



Figure 1. Equipment.

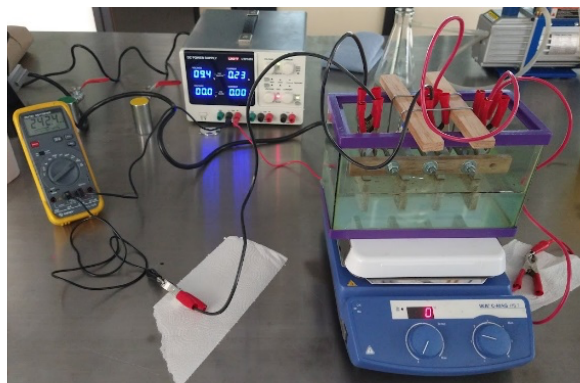


Figure 2. Electrode arrangement and electrocoagulation process.

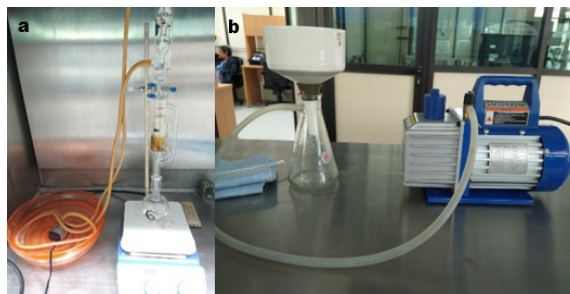


Figure 3. (a) Extraction system, (b) Filtration system.

After adding the supporting electrolyte water, its conductivity was measured using a conductometer with 2% accuracy according to the characteristics described in the Mexican legislation in force NMX-AA-093-2018 for conductivity measurement of any kind of water. This conductivity measurement allowed the determination of voltage demand to perform the electrocoagulation treatment.

After the wastewater analogue was treated, the lubricant oil waste flocs were separated by a vacuum filtration using an economical filter paper of a 1 μ m pore diameter. This filtration method was done by using a Büchner funnel and a vacuum flask. The vacuum was provided by a 0.5 HP vacuum pump and the flocs were captured on a 1 μ m pore filter paper that was later dried in a muffle furnace of 1500 W to eliminate all the humidity and prepare it for recovered lubricant oil waste quantification.

The percentage of recovered lubricant oil waste was determined by a quantitative method named Soxhlet extraction as displayed in Figure 3 (a). This technique used an organic solvent to extract the recovered lubricant oil waste captured on the dried filter paper. The organic solvent selected for the extraction was hexane which was heated and recirculated until the filter paper changed its yellowish coloration to white.

Once the extraction concluded, the obtained product was a liquid mixture containing hexane and lubricant oil waste. This homogeneous mixture was separated by a micro scale distillation stage shown in Figure 3 (b). The distilled corresponded to the recovered hexane and the residue was the lubricant oil waste recovered from the electrocoagulation treatment. This residue was weighted in an analytical scale to determine the quantity of lubricant oil waste recovered after the electrocoagulation treatment.

2.2 Electrodes cleaning

The aluminum 6061 electrodes were prepared before their introduction to the electrochemical cell. This preparation consisted in two cleaning steps. The first step included a mechanical cleaning using soapy potable water and a synthetic fiber to eliminate impurities, as well as a consistent polish with sandpaper. The second step involved the use of distilled water and ethanol. The distilled water helped to rinse the dissolved salt contained in the soapy potable

water while the ethanol promoted the elimination of residual lubricant oil waste adhered to the electrodes after the electrochemical treatment.

It is noteworthy that the chemical cleaning did not involve the use of strong acids neither alkali since, according to IKA, 2021, they could promote quick degradation of electrodes resulting in a frequent replacement which means higher operational costs.

2.3 Wastewater analogue

A wastewater analogue was prepared adding approximately 1 ml of lubricant oil waste to 1 liter of potable water contained in the rectangular batch reactor. Right after the lubricant oil waste addition, the wastewater analogue was mixed at 260 revolutions per minute until the electrocoagulation process finished. It is worth noting that the design of a wastewater analogue for the experimental studies facilitated removal quantification and determination of the electrocoagulation treatment efficiency in the proposed system.

The lubricant oil waste utilized for the wastewater analogue was donated by an engine workshop therefore, waste characteristics and properties were difficult to determine since it was a mixture of different lubricant oils. Nevertheless, Fong *et al.* (2017) states that the average density of used lubricant oils is 911 mg/L. This value was considered to establish the quantity of lubricant oil waste that needed to be added to the potable water matrix aiming to achieve a concentration above the authorized limit of 100 mg/L of oils contained in domestic wastewater that is discharged to the sewer as defined in Mexican legislation NOM-002-SEMARNAT-1996.

2.4 Electrocoagulation parameters

The electrocoagulation process is controlled by three main parameters: current density, water conductivity and operation time (Tahreen *et al.*, 2020). These parameters were manipulated during the experimental study according to the lubricant oil waste characteristics aiming to get the major efficiency. Each parameter has different influence in the electrocoagulation process.

In case of current density, it determines electrodes liberation speed during the electrochemical reaction that produces hydroxymetalic compounds that promote coagulation of oily species. In addition, it contributes to water turbidity decrease. On the contrary, water conductivity is related to energy consumption during the electrochemical process. If conductivity is increased, consumed voltage decreases. Hence, the more conductive water is, the less energy supply is required, and costs are reduced.

Aiming to control wastewater analogue conductivity, a supporting electrolyte was added to half of the trials. The selected supporting electrolyte was a 2g/L sodium chloride solution because of its availability, and low cost. By adding 100 ml of this electrolyte solution water, conductivity electrons transference was promoted, and electrocoagulation effectiveness was increased (Perren, *et al.*, 2018).

The concentration of the supportive electrolyte was selected based on Perren, *et al.*, 2018 investigation which results showed that 2g/L of a NaCl solution were viable enough in economic terms to achieve significant removal results.

It is worth to mention that the main difference between operating conditions on Table 1 and Table 2 is the usage of the supporting electrolyte that modifies the conductive character of the wastewater analogue to promote electrocoagulation process.

Table 1. Operating conditions without supporting electrolyte.

Operating conditions without supporting electrolyte					
Test	pH	Conductivity ($\mu\text{S}/\text{cm}$)	Voltage (V)	Current intensity (mA)	Operating time (min)
1	6.97	2566	9.55	240	10
2	8.1	2180	11.86	240	20
3	8.18	2182	11.86	240	30
4	6.96	2182	11.86	240	40
5	8.15	2328	11.57	240	50

Table 2. Operating conditions with supporting electrolyte.

Operating conditions with supporting electrolyte					
Test	pH	Conductivity ($\mu\text{S}/\text{cm}$)	Voltage (V)	Current intensity (mA)	Operating time (min)
1	7.7	3048	8.3	240	10
2	7.7	3056	8.4	240	20
3	8.1	2920	8.7	240	30
4	8.1	2896	8.7	240	40
5	7.7	3142	8.2	240	50

Regarding the operation time, experimental trials were done increasing process time 10 minutes. This increase kept the operation time between a range of 10 to 50 minutes allowing the production of hydroxymetalic compounds in an enough quantity to achieve a considerable lubricant oil waste removal according to Faraday's Law that establishes that mass formed by an electrolytic reaction is proportional to the operation time.

2.5 Electrocoagulation treatment

Electrocoagulation treatment started with the cleaning of aluminum 6061 electrodes. After, electrodes cleaning, batch reactor was filled with 1 L of potable water. Potable water initial conductivity and pH were measured and registered as a reference to compare them with the final values. Following the measurement of initial parameters, supporting electrolyte was prepared and added to 50% of experimental trials.

Subsequently, 1 ml of lubricant oil waste was weighted and added directly to potable water. Also, a pair of magnetic stirrers were introduced to the batch reactor to obtain a homogeneous mixture. Stirring was turned on at 260 revolutions per minute and did not stop until electrocoagulation treatment finalization.

Succeeding water analogue preparation, electrodes arrangement was introduced to the batch reactor and connected to the DC power supply. Then, operation parameters were set up, system was energized, and process time started to count.

After the lapse of operation time, DC power supply was turned off to deenergize the experimental system. Electrodes arrangement was disconnected and disassembled. Treated water was separated from flocs using vacuum filtration and preserved in glass beakers with a capacity of 500 ml to allow flocs sedimentation and facilitate quantification of removed oil waste.

2.6 Removed oil waste quantification

The quantification of removed lubricant oil waste is based on the NMX-AA-005-SCFI-2013 that establishes the method to determine the concentration of oils in any type of water. This method is merely gravimetric since it does not classify oil's physicochemical characteristics.

The quantitative method uses Soxhlet extraction to recover lubricant oil waste removed after electrocoagulation treatment. The flocs were trapped in the filter paper due to vacuum filtration. This filter paper was placed into the Soxhlet extractor and assembled in a round bottom flask filled with 40 ml of hexane.

The organic solvent is boiled and condensed to be cycled through the syphon and the side arm to collect the lubricant oil waste. The hot solvent with the lubricant oil waste falls to the base of the round bottom flask. Subsequently, the mixture is distilled at 103°C using a microscale equipment. The hexane was condensed and reused for the following trials?

extraction. The removed lubricant oil waste was recovered on the base of the round bottom flask of the distillation equipment.

The bottom flask of the distillation equipment with the lubricant oil was weighted, which was registered and compared with the initial quantity of lubricant oil waste added to the analogue wastewater.

2.7 Treated water quality analysis

The quality of the treated water was analyzed after removed lubricant oil quantification. The analysis was practiced aimed to determining if the treated water was suitable for domestic use, crops irrigation or to be purified to use it as drinking water. The analysis included the determination of indicators that define the quality of water. These indicators are pH, alkalinity, turbidity, color, conductivity, total hardness, total suspended solids, total dissolved solids, and the presence of chlorides.

Aiming to verify the viability and reliability of the proposed electrochemical treatment, a comparative analysis of three samples of water: drinking water used for the wastewater analogue preparation, treated water, and wastewater analogue, was performed. The comparative analysis was performed by *COMPAÑÍA MEXICANA DE AGUA, S. A. DE C. V.*

3 Results and discussions

To determine the removal effectiveness of the proposed treatment technique, the total amount of lubricant oil removed from the wastewater analogue was estimated for each of the ten experimental tests. This process was performed according to the gravimetric method described on NMX-AA-005-SCFI-2013. Overall, the collected data was used for performing a direct comparison between the amount of lubricant oil waste, contained within the wastewater analogue matrix, before and after the application of electrocoagulation process and the vacuum filtration. Tables 3 and 4 report the removal effectiveness, as a percentage of the initial amount of lubricant oil waste removed, for all the test.

Table 3. % Recovery of oil without support electrolyte.

Test	Oil recovery with supporting electrolyte		
	$m_{initial}$	$m_{recovered}$	% Recovery
1	0.8225	0.3515	43%
2	0.6867	0.593	86%
3	0.6867	0.5472	80%
4	0.8225	0.5452	66%
5	0.8754	0.387	44%

Table 4. % Recovery of oil with support electrolyte.

Test	Oil recovery with supporting electrolyte		
	$m_{initial}$	$m_{recovered}$	% Recovery
1	0.6867	0.5583	81%
2	0.6867	0.5847	85%
3	0.6867	0.6225	91%
4	0.6867	0.5748	84%
5	0.6867	0.5668	83%

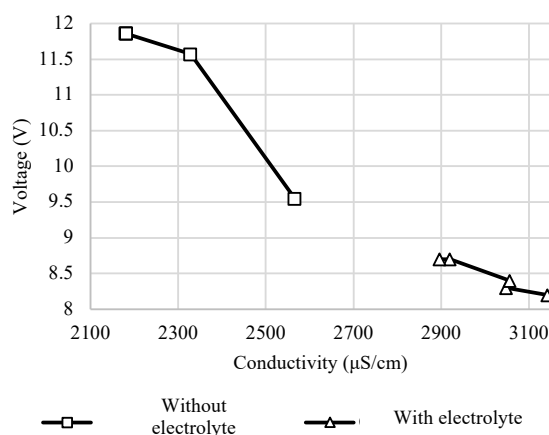


Figure 4. Behavior of the voltage with respect to the increase in conductivity of the medium.

According to the experimental results, removal effectiveness of 86% was achieved without the usage of any supportive electrolyte solution. Nevertheless, the largest removal effectiveness reached up to 91% under the optimum operating conditions within the value ranges that were proposed for each of the critical parameters analyzed: a fixed current density of A/m^2 , water conductivity varying from 2180 to 3142 ($\mu S/cm$) and an operation time within the range from 10 to 50 minutes. The optimum conditions within the range analyzed were an operating time of 30 minutes, a current density supply of $40 A/m^2$ and addition of 100 mL of a 2 g/L NaCl solution as an electrolyte additive.

The characterization trials performed with the addition of an electrolyte additive produced higher removal effectiveness than the tests performed in the system without the addition an electrolytic solution, due to an easier formation of hydroxymetalic species in the anode induced by a higher conductivity of the medium, also allowing more flocs creation. This is valid in the studied range of

conductivity, the effects on higher conductivity values is out of the limits of this study. Furthermore, the graph shown in Figure 4 notes that the energy supply was reduced due to a higher conductivity that decreased system resistance. Also, a lower voltage value was required to operate the electrocoagulation process. Despite these positive effects of the addition of the electrolytic solution to the wastewater, it is worth reporting that the more salt content in the water is, the more difficult is the further vacuum filtration process.

It is worth making a focused analysis over the adequate salt concentration (therefore conductivity) range that allows to operate the system as efficiently as possible, with the lowest energy supply and wastes, as long as it allows a mild filtration subsequent stage.

The further analysis of the quality parameters of a sample of clean water, treated water and wastewater analogue allowed to directly measure the deviation of the properties of the treated water while comparing it with the base water, but also to determine the viability of the electrochemical treatment. The results are reported in Table 5.

The treatment process allowed to recover the clarity and colorless characteristic of the treated water almost completely, as it is shown by the color parameter measurement. This is a great advantage over the coloured wastewater analogue sample, which was perceived deeply coloured. In addition, the turbidity is another property related to water clarity, and it was possible to return treated water to a value close to the starting point through electrocoagulation that produced a water matrix with a medium quality.

Regarding the pH variation of the treated water, it is worth noting that it remained practically the same as potable water sample measure. Throughout the operation, the system tended to a slightly acid character, with a total decrease of less than 0.1 pH units. This is a desired effect because it allows the pH to remain in a very close range to the potable water.

The total hardness of the water that includes all of the dissolved calcium and magnesium compounds, is an indicator over which electrocoagulation had a positive effect. The results demonstrate that the process promotes an easier separation of these compounds through flocs filtration. The treated water total hardness decreased in 34 mg per liter. This is an interesting aspect of electrocoagulation technique, as long as it would contribute to the decrease of solid deposits in clog pipes.

Table 5. Water analysis results (a).

Sample	pH	Turbidity (NTU)	Color (Pt/Co)	Chlorides (mg/L)
Base water	8.44	1.077	0.00	96.96
Treated water	8.41	0.97	14.33	190.94
Untreated water	8.44	25.35	398.63	94.97

In contrast, the total content of dissolved solids in the treated water is the most inconvenient parameter of the treated water, as it raises up to 519 mg/L, in comparison to the 408 mg/L value of the potable water and the wastewater's 468 mg/L measurement. Therefore the total content of suspended solids and the system electric conductivity presented the same behaviour due to the continuous generation of ionic species throughout the process and the lack of a post treatment phase that made possible their capture.

Throughout the experimental trials, the water filtration was made with an economical filter paper of a 1 μ m pore diameter, therefore it is paramount to design an adequate filtration system that allows to get rid of the newly generated dissolved solids as long as this excess may lead to issues related to a poor quality of the treated water.

Finally, a technical and economical thoroughly evaluation must be done over the viability of the system operation considering the energy supply, the setting-up and maintenance of an electrocoagulation reactor and the optimum geometry and electrodes arrangement.

Conclusions

The results demonstrate that the application of electrocoagulation technique as a treatment method to remove industrial lubricant oils contained in wastewater is an effective alternative that allows to easily remove the oil waste from the polluted water system. It is a treatment process which application seems viable due to the mild operating conditions, the affordable elements and equipment that make up the system, the lack of additional additives that may require additional though separation stages and the properties achieved in the wastewater after the treatment.

Within the established operating conditions range of values, the largest industrial lubricant oil removal effectiveness was up to 91% when the wastewater presented a pH value of 8.1 units and a conductivity of 2920 μ S/cm reached with the addition of 100 mL of a 2 g/L NaCl electrolytic solution. Furthermore, the system used two pair of aluminum 6061 electrodes, that were supplied with a current density of 40 A/m² throughout an operating time of 30 minutes.

Finally, the electrocoagulation technique does not develop hazardous properties in the treated wastewater, but it leads to recover most of the characteristics of the clean potable water. The effect over the pH is virtually meaningless; a good appearance of the wastewater is achieved due to a low value of remaining turbidity and color. Even the process has a positive effect over the hardness of the water and the alkalinity, which diminishes after the treatment process to a lower value than the potable water; nevertheless, this treatment method without a proper ion exchange filtration stage remains with a higher concentration

of dissolved solids and chlorides. This promising evaluation allows treated water further usage such as service or irrigation water.

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